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IMPROVED IMPACT FRACTURE RESISTANCE IN OXIDATION-TOUGHENED Si_3N_4–20 vol% ZrO_2

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December 1979

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Prepared for:

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

When impacted by 1.2-mm-diameter tungsten carbide spheres at velocities to 200 m/s, oxidized Si_3N_4-20 vol% ZrO_2 exhibited decidedly less fracture damage than unoxidized material. The impact velocity necessary to initiate ring and radial cracks was significantly higher for oxidized materials, and the rate at which fracture damage, once nucleated, developed was substantially lower. This enhanced dynamic performance is consistent with the quasi-static properties reported by Lange and may involve oxidation-enhanced quasi-static properties reported by Lange and may involve oxidation-induced compressive surface stresses, oxidation-induced softening, or both.

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Additional experiments are recommended to confirm and further investigate these promising results.

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Al2O3

INTRODUCTION

Si3N4-ZrO2

In a current ONR-sponsored research program at Rockwell International, Dr. F. F. Lange is using an oxidation technique to induce compressive stresses in the near-surface regions of Si_3N_4-ZrO_2 specimens.[1] Silicon nitride powder is first thoroughly mixed with about 20 vol% zirconium oxide and 4 vol% Al_2O_3 powders, then hot pressed to achieve a fully dense plate. The plate is then given a subsequent anneal at 700°C for 5 hr in air. The zirconium oxynitride within several grain diameters of the surfaces is oxidized to the monoclinic form with an accompanying increase in molar volume of about 4-5%. The expansion of the lattice in the surface regions gives rise to substantial compressive stresses on the surface (and significant tensile stresses in the interior).

Lange's initial investigations of mechanical properties of this surface-strengthened material gave promising results. Diamond pyramid hardness tests and notched four-point bend tests gave increases of 20% and 25% in apparent surface toughness and flexural strength, respectively. These results encouraged us to investigate the response of this material to particle impact.

Using 1.2-mm-diameter WC spheres, we performed a series of impact experiments at various velocities on oxidized (surface-strengthened) and unoxidized Si_3N_4-ZrO_2 to determine the damage phenomenology, the threshold velocities for ring and radial cracks, and the rates of impact damage development and thus evaluated the effects of the oxidation treatment on resistance to particle impact damage. This report presents the results of this study.

MATERIALS, EXPERIMENTS, AND RESULTS

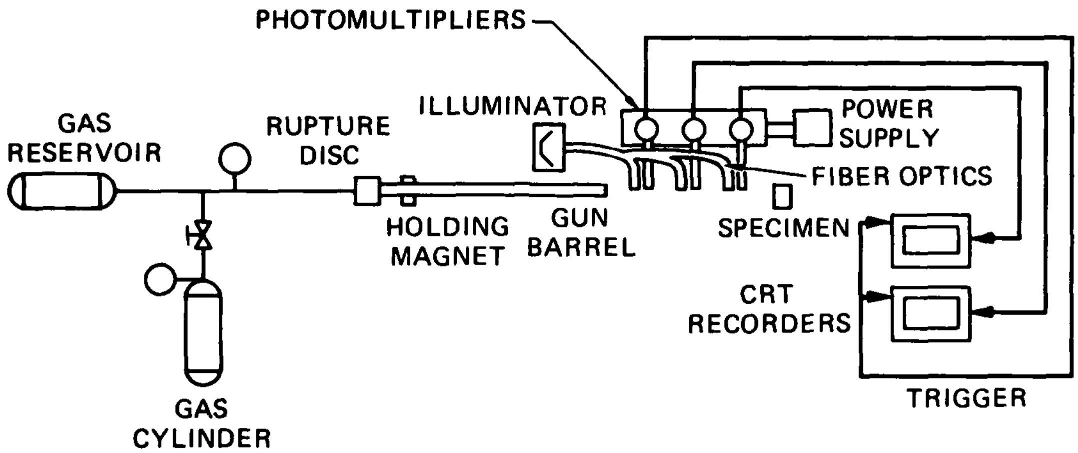
Two specimens each of oxidized and unoxidized Si_3N_4-20 vol%ZrO_2 were provided as halves of broken 4-point-bend specimens by Dr. F. F. Lange. Nominal dimensions were 3 x 6 x 18 mm. The surface finish of the unoxidized specimens was improved by polishing to make it comparable to the surface of the oxidized specimens.

The specimens were impacted with 1.2-mm-diameter tungsten carbide spheres at various velocities using the compressed air gun shown in Figure 1. Discs of various materials and thicknesses ruptured at various air pressures and allowed for a wide range of particle velocities. Velocities were measured with a photomultiplier arrangement.

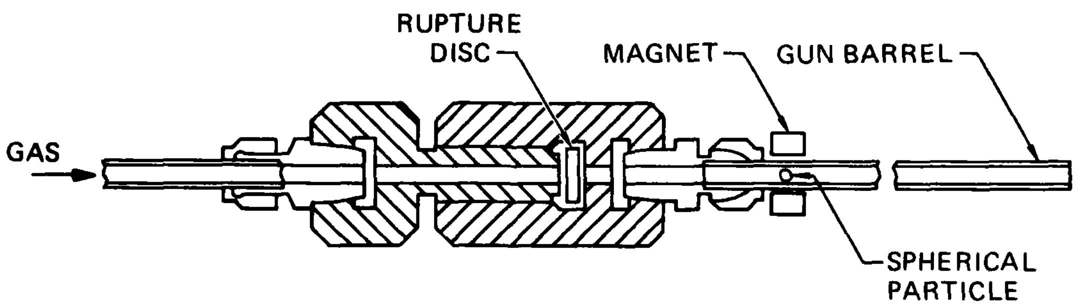
Table I summarizes the conditions and results of the experiments. A range of impact velocities and specimen damage was obtained in each material. At the lowest velocities, no damage could be detected at magnifications up to 400X. Slightly higher velocities produced a plastic impression, and at higher velocities, ring cracking then radial cracking began.

The size of the plastic impression increased monotonically with increasing impact velocity. A profilometer, used to measure the shapes of the impression, verified that plastic flow at the impact sites occurred at velocities well below those necessary for ring crack formation.[2] The depths of the plastic impressions are recorded in Table I. Deeper impressions were sustained by oxidized material impacted at similar velocities as unoxidized specimens, showing that the oxidation treatment decreased the dynamic hardness.

A similar effect on quasi-static hardness was found in a series of diamond pyramid hardness indents at loads ranging from 0.1 to 20 kg. The average hardness of the oxidized material was 1200 kg/mm^2 compared with a value of 1600 kg/mm^2 for unoxidized specimens. A more pronounced softening in the surface layers of the oxidized specimens was indicated by the hardness results (1000 kg/mm^2) at 0.1 and 0.2 kg indenter loads.



(a) SCHEMATIC OF THE FACILITY



(b) DETAIL OF RUPTURE DISC ASSEMBLY FOR FAST RELEASE OF GAS

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FIGURE 1 PARTICLE IMPACT FACILITY

Table I

PARTICLE IMPACT EXPERIMENTS IN Si_3N_4-20 vol%ZrO_2

UNOXIDIZED SPECIMENS*†					OXIDIZED SPECIMENS δ‡				
Test Number	Impact Velocity (m/s)	Depth of Plastic Impression (μm)	Ring Crack Diameter ($\times 10^2$ cm) inner d_i	Ring Crack Diameter ($\times 10^2$ cm) outer d_o	Test Number	Impact Velocity (m/s)	Depth of Plastic Impression (μm)	Ring Crack Diameter ($\times 10^2$ cm) inner d_i	Ring Crack Diameter ($\times 10^2$ cm) outer d_o
U-10*	16.9	0.19	1.9	1.9	0-09δ	34	0.95	-	-
U-03*	19.2	0.32	2.2	2.2	0-10δ	39	0.98	-	-
U-15*	22.0	na	2.4	2.4	0-12δ	42.3	1.8	-	-
U-14*	24.2	na	2.2	2.4	0-11δ	46.2	1.9	3.0	3.0
U-06*	28.6	0.70	2.3	2.5	0-08δ	60	2.7	3.5	3.7
U-08*	33.8	0.92	2.2	2.9	0-01δ	85	3.6	3.1	4.0
U-07*	38.3	0.98	2.1	2.8	0-04δ	120	3.8	3.2	4.3
U-04*	44.1	0.95	2.2	3.1	0-16‡	144	na	3.9	4.8
U-05*	48.4	1.3	2.2	3.3	0-15‡	169	3.4	3.3	6.0
U-02*	50.8	1.6	2.0	3.3	0-13‡	195	6.4	2.8	6.5
U-17*	79.4	2.3	2.1	4.4	0-14‡	195	5.9	?	6.5
U-18*	108	3.0	2.3	5.1					
U-19*	121	3.3	2.1	5.3					
U-29†	145	na	2.1	5.7					
U-30†	159	na	2.1	6.8					
U-21†	181	3.6	2.1	6.9					
U-20†	282	5.2	1.6	7.0					

Rockwell International Specimen Designation

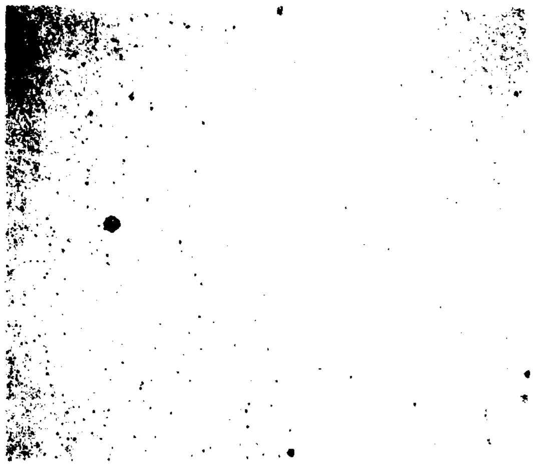
*Specimen R12.16.88 MMS 6.26 78.6
†Specimen R12.16.77 MMS 6.26 78.8
δSpecimen R12.16.771 MMS 6.26.78.1
‡Specimen R12.16.771 MMS 6.26.78.3

Figure 2 compares the fracture damage produced on the surface of unoxidized and oxidized specimens at an impact velocity of 45 m/s. A portion of a single ring crack was produced in the oxidized material, whereas several well-developed ring cracks were produced in the unoxidized specimens. Ring cracks became more numerous as velocity increased. Figure 3 shows the damage produced in the two materials by 145 m/s impact. An annulus of cracked material developed about the center of the impact site. The annular area was always greater in the unoxidized material. At a velocity of 180 m/s, radial cracks appeared in the unoxidized material, Figure 4(a). However, no radial cracks were observed in oxidized material at velocities up to 195 m/s, Figure 4(b). The halo that is evident in Figures 3 and 4 is shallow surface damage produced when the tungsten carbide sphere fragments and impacts the surface. Tungsten carbide spheres break at velocities of about 100 m/s and greater when impacted against these specimens.

Several impact sites in oxidized and unoxidized material were sectioned, and the section surfaces were polished and examined with a microscope to determine the cracking patterns in the specimen interior. Both materials showed the Hertzian cone-shaped cracks.

The threshold conditions for fracture damage were substantially higher for the oxidized material, Table II. Ring cracking first appeared in unoxidized material at an impact velocity of 17 m/s; velocities of 46 m/s, however, were required to produce ring cracks in the oxidized material. Similarly, radial cracks were observed at 180 m/s in unoxidized material, but were not produced in oxidized material at 195 m/s.

The outer radius of the annular area containing ring cracks increased monotonically with velocity in both materials, but the inner radius remained roughly constant. Figure 5 shows that the inner radius of the cracked zone is larger for the oxidized material and that the outer radius is smaller. Thus, for any given impact velocity, the damaged area in oxidized material is significantly smaller than in the untreated material.



UNOXIDIZED

OXIDIZED

200 μm

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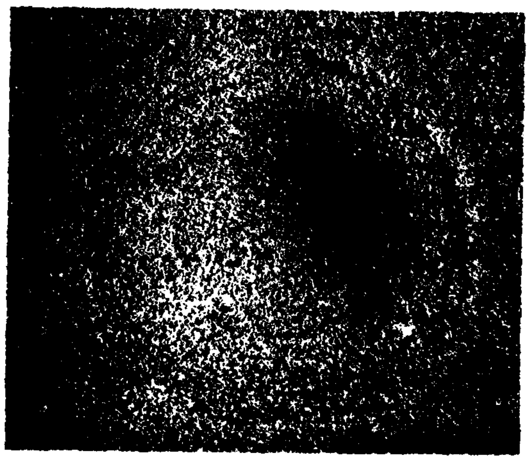
FIGURE 2 SURFACE DAMAGE PRODUCED BY PARTICLE IMPACT AT 45 m/s

UNOXIDIZED



OXIDIZED 200 μm

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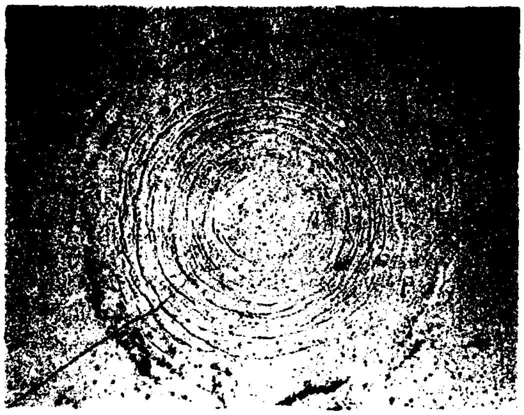
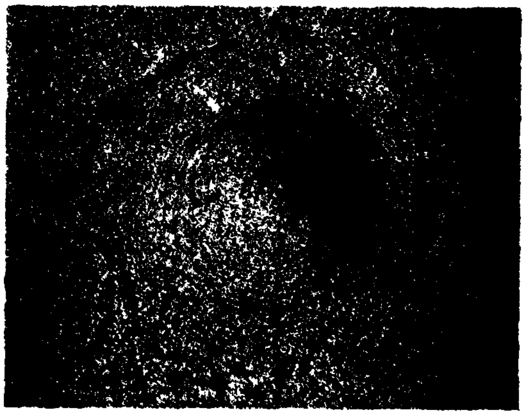
FIGURE 3 SURFACE DAMAGE PRODUCED BY PARTICLE IMPACT AT 145 m/s



(a) UNOXIDIZED — 180 m/s



200 μm

(b) OXIDIZED — 195 m/s

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FIGURE 4 SURFACE DAMAGE PRODUCED BY PARTICLE IMPACT AT 180 AND 195 m/s

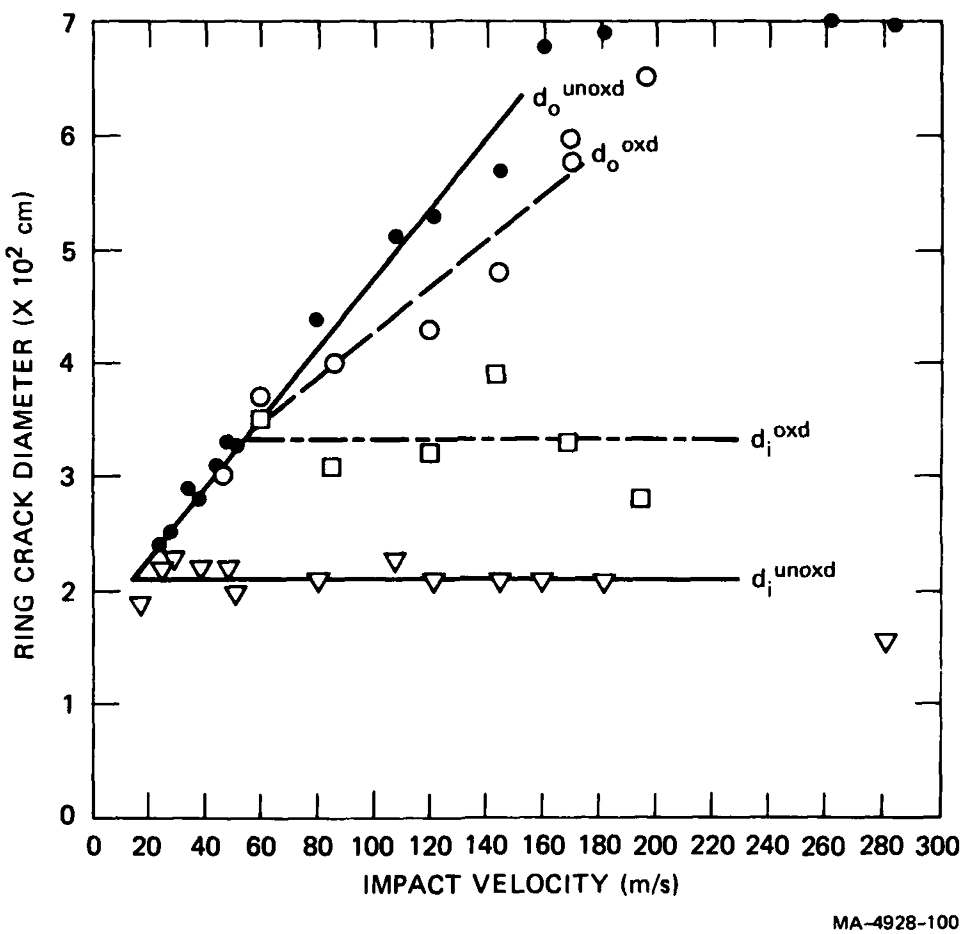

FIGURE 5 GROWTH OF DAMAGE ZONE WITH IMPACT VELOCITY FOR OXIDIZED AND UNOXIDIZED Si_3N_4-ZrO_2

Table II

THRESHOLD VELOCITIES FOR IMPACT DAMAGE IN Si_3N_4-20% ZrO_2

	Oxidized	Unoxidized
Ring crack threshold (m/s)	46	17
Radial crack threshold (m/s)	>195	180

DISCUSSION

The impact experiments were performed to see if oxidizing treatments, which enhanced quasi-static properties, would also enhance particle impact resistance. The material used here was probably not optimal; other compositions and oxidation treatments could be expected to exhibit greater dynamic fracture resistance.

The experimental results indicated a clear superiority of the oxidized material in resisting damage from an impacting particle and suggest the usefulness of this treatment in applications where incipient fracture constitutes failure, e.g., laser windows. Multiple impact studies are required to ascertain the benefit of oxidation in applications where mass loss and gross thinning (erosion) of a component constitute failure. The use of oxidized material in high temperature oxidizing environments, such as encountered by turbine blades, appears of doubtful benefit, however, because previous impact tests indicate that over-oxidation enhances erosion rates.[3]

The observed enhancement of impact damage resistance could be attributable to oxidation-induced surface compressive stresses or to oxidation-induced softening or to both. Quasi-static hardness tests indicate a decrease in hardness in oxidized materials. Likewise, a decrease in the dynamic hardness, as inferred from measurements of the plastic impression depth (Table I) with a profilometer, was produced by the oxidation treatment. However, the important aspect of the impact damage resistance is in the fracture propensity of the two materials. Ring cracks and radial cracks initiated at lower levels and developed at more rapid rates in the unoxidized material, which suggests that the oxidation-induced surface compressive stresses inhibit and suppress fracture activity.

Additional experiments should be performed with particles of other sizes, shapes, materials, angles of incidence, and velocities to confirm

the promising observations of the present work. Tests at higher velocities are desirable to ascertain whether residual tensile stresses in the interior of the surface-strengthened material lead to poorer impact resistance in other velocity ranges. Companion experiments on conventional Si_3N_4 should be performed to establish the relative behavior of $Si_3N_4 \cdot 20\% \ ZrO_2$.

The mechanism responsible for improved impact resistance should be established. In particular, the roles of oxidation softening and transformation-induced surface compressive stresses should be examined to ascertain whether the production of surface compressive stresses is a useful avenue for obtaining more erosion-resistant ceramics.

CONCLUSIONS

Compressive surface stresses do not alter the phenomenology of particle impact damage in Si_3N_4-20% ZrO_2. Plastic flow, ring cracking, cone cracking, and radial cracking occur, in that order, in oxidized (surface-strengthened) as well as unoxidized material. Under given impact conditions, however, the oxidized material sustains decidedly less fracture damage than the unoxidized material (in the particle size, shape, type, and velocity range particular to these experiments). The impact velocity necessary to initiate fracture is significantly higher for oxidized material, and the rate at which the damage zone, once initiated, develops is substantially lower. Thus, enhanced dynamic performance is consistent with the enhancement of quasi-static properties found by Lange.

The results suggest that transformation-induced surface stresses may be helpful in improving the resistance of ceramic components to damage and erosion from particle impact. Additional experiments with particles of other sizes, shapes, types, angles of incidence, and velocities should be performed to confirm and further investigate these promising initial results.

REFERENCES

1. F. F. Lange, "Compressive Surface Stresses Developed in Ceramics by an Oxidation-Induced Phase Change," Technical Report No. 5, submitted to Office of Naval Research, on Contract N00014-77-C-0441 (July 1978).

2. This behavior is opposite that observed by A. G. Evans, U.C. Berkeley (private communication), and considerations of the stress distribution about a plastic contact show that radial cracks indeed should be expected because of large circumferential tensile stresses. Elastic contact produces large stresses in the radial direction and hence encourages ring cracking. In the elastic-plastic regime, however, the stress distribution may favor ring or radial cracks and thereby account for the discrepancy in observed fracture behavior.

3. K. Ç. Dao, D. A. Shockey, L. Seaman, D. R. Curran, and D. J. Rowcliffe, "Particle Impact Damage in Silicon Nitride," Annual Report Part III to Office of Naval Research, Arlington, VA, on Contract N00014-76-C-0657 (1979).